K.03
5/16/95

CRA

CONESTOGA-ROVERS & ASSOCIATES
O'Hare Corporate Towers One
10400 W. Higgins Road, Suite 103
Rosemont, Illinois 60018
(708) 299-9933 Fax: (708) 299-6421

June 2, 1995

Reference No. 2372

Mr. Anthony Rutter
Director, Waste Management Division
Remedial Project Manager
U.S. Environmental Protection Agency
77 West Jackson Boulevard
Chicago, Illinois 60604

Mr. Regan S. Williams
State Project Coordinator
Ohio EPA - Division of
Emergency & Remedial Response
2110 East Aurora Road
Twinsburg, Ohio 44087

Gentlemen:

Re: Revisions to Quality Assurance Project Plan
Operation, Maintenance & Monitoring
Summit National Superfund Site
Deerfield, Ohio

Further to the August 26, 1994 approval of the Quality Assurance Project Plan (QAPP) of the Operation, Maintenance and Monitoring Plan for the Summit National Superfund Site in Deerfield, Ohio by the United States Environmental Protection Agency (USEPA) and the Ohio Environmental Protection Agency (OEPA), the laboratory approved for analyses of air samples no longer analyzes air samples. Conestoga-Rovers & Associates, on behalf of the Summit National Facility Trust (SNFT), request USEPA and OEPA approval of Quanterra, Incorporated of City of Industry, California for analyses of air samples. Enclosed you will find revisions to the QAPP to change the laboratory used for air analyses, including two copies of the revised QAPP pages, laboratory Standard Operating Procedures and the organizational figure for approval by USEPA and OEPA.

Should you have any questions, please do not hesitate to contact the undersigned.

Yours truly,

CONESTOGA-ROVERS & ASSOCIATES

Nancy M Bergstrom for

Steve Whillier, B. Sc.

SW/ev/207
Encl.

c.c. Richard McAvoy
Gary Gifford
Patrick Steerman
Kenneth Walanski
Jack Michels
Steve Hayle
Nancy Bergstrom

Terri Wynnik - Sample Custodian - NUS

- receive and inspect the incoming sample containers
- record the condition of the incoming sample containers
- sign appropriate documents
- verify chain of custody and its correctness
- notify Project manager of sample receipt and inspection
- assign a unique identification number and customer number and enter each into the sample receiving log
- with the help of the operations manager, initiate transfer of the samples to appropriate lab sections
- control and monitor access/storage of samples and extracts

Quanterra, Incorporated (Quanterra)
18501 East Gale Avenue, Suite 130
City of Industry, California 91748
(818) 965-1006

- as subcontractor to NUS will perform the analysis of VOC in air using method TO-14

Paul Henige - Project Manager - Quanterra

- ensures all resources of the laboratory are available on an as-required basis
- overview of final analytical reports
- approval of the QAPP

Val Mallari - Operations Manager - Quanterra

- coordinate laboratory analyses
- supervise in-house chain-of-custody
- schedule sample analyses
- oversee data review
- oversee preparation of analytical reports
- approve final analytical reports prior to submission to the Engineering Consultant

Scott Hoatson - QA Officer - Quanterra

- overview laboratory quality assurance
- overview QA/QC documentation
- conduct detailed data review
- decide laboratory corrective actions, if required
- technical representation of laboratory QA procedures
- preparation of laboratory SOPs
- approval of the QAPP

Jan Dalman - Sample Custodian - Quanterra

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Primary responsibility for project quality rests with CRA's QA/QC Officer - Analytical and Field Activities. Ultimate responsibility for project quality rests with CRA's Project Manager. Independent quality assurance will be provided by the Laboratory Project Manager and QA Officer prior to release of all data to the contractor.

USEPA RESPONSIBILITIES

The USEPA Region V Remedial Project Manager (RPM) will be responsible for the overview of this project. The RPM will also be responsible for providing approval of the QAPP. Anthony Rutter is the RPM for the Remedial Action activities.

12.3 QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT DATA

The overall QA objective is to develop and implement procedures for field sampling, chain-of-custody, laboratory analyses and reporting that will provide results which are legally defensible in a court of law. Specific procedures for sampling, chain-of-custody, laboratory instruments calibration, laboratory analysis, reporting of data, internal quality control, audits, preventive maintenance of field equipment and corrective action are described in other section of this QAPP. The purpose of this section is to address the specific objectives for accuracy, precision, completeness, representativeness and comparability.

12.3.1 Level of QC Effort

To assess the quality of data resulting from the field sampling program, field duplicate samples, field blank samples (bailer rinse), trip blank samples, preservative blank samples and matrix spike samples will be taken and submitted to the analytical laboratory.

Field duplicate samples will be collected at a frequency of one per ten or fewer investigative samples per parameter set for all sample matrices, with a minimum of one field duplicate sample submitted per sampling event. Matrix spike and matrix spike duplicate (MS/MSD) samples will be analyzed at a minimum frequency of one per 20 or fewer samples for each organic analysis. Laboratory spikes/laboratory spike duplicates (LS/LSD) will be analyzed at a minimum of one per 20 or fewer samples for air samples. For the metals analyses, one matrix spike and laboratory duplicate (MS/DUP) or MS/MSD will be analyzed at a minimum frequency of one per 20 or fewer investigative samples.

Field blank samples will be submitted at a frequency of one per ten well purging/sampling equipment cleanings or a least once per day of well purging/sampling equipment cleanings, whichever is more frequent. Field blank samples will be collected by routing deionized water through decontaminated sampling equipment. Field blank samples will be analyzed to check procedural contamination and/or ambient conditions and/or sample container contamination at the Site that may cause sample contamination.

Preserved trip blank samples for VOC analyses (prepared by the laboratory and consisting of organic-free water poured into the sample vials) will be shipped with each shipping container of VOC sample vials by the laboratory. Trip blank samples will be handled in a manner consistent with actual field sample handling and will be shipped back to the laboratory with the monitoring well samples. The trip blanks will provide a measure of potential cross contamination of samples during shipment and handling. However, it should be noted that trip blanks will not be opened in the field.

Upon examination of the results obtained by the laboratory, if any of the aforementioned blanks are found to contain any of the target analytes, the following procedure will be followed. First, determine if the contamination is real by examining the associated investigative samples and method blanks. If the contamination can be traced to an isolated source, e.g. a highly contaminated sample, the data is to remain unqualified. Otherwise, the data will be examined to determine the extent of contamination and all associated data will be qualified according to the data validation guidelines given in Section 12.9.

Field duplicate samples will be analyzed to check for sampling and analytical reproducibility. Field duplicate samples will be used as a measure of precision throughout the sampling event. Comparison of field duplicate samples will be based upon the target analytes, both non-detected and detected, and the relative percent differences (RPD) of each

analyte's concentrations. The parameters which do not meet the criteria may only be used as qualitative measurements. Professional judgment shall determine the RPD limits on a sample-to-sample basis.

The level of QC effort provided by the laboratory for analysis of the samples will be equivalent to the level of QC effort specified in the standard operating procedures (SOPs) in Appendix 12.1.

The level of QC effort for the field measurements of pH and specific conductance will be as described in the SOPs in Appendix 12.1. Temperature readings will be obtained with pH measurements. Water level measurements will be to the nearest 0.01 ft. using an electric sounding water level meter.

12.3.2 Accuracy, Precision and Sensitivity of Analyses

The fundamental QA objective with respect to accuracy and precision of laboratory analytical data is to achieve the QC acceptance criteria of the analytical protocols. The sensitivities required for the analyses will be at least the targeted quantitation limits in Tables 12.3 through 12.6. It should be noted that the quantitation limits listed are targeted quantitation limits. Actual sample quantitation limits are highly matrix dependent.

SOPs for laboratory analyses are provided in Appendix 12.1. These include the required accuracy, precision, sensitivity of the analyses. SOPs for the field equipment to measure pH, conductivity and temperature are also provided in Appendix 12.1.

12.3.3 Completeness, Representativeness and Comparability

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions. It is expected that the project laboratory will provide data meeting the QC acceptance criteria for 90 percent or more for all samples tested using the referenced methods. Following completion of the analytical testing, the percent completeness will be calculated by the following equation:

$$\text{Completeness (\%)} = \frac{\text{Valid Data Obtained}}{\text{Total Data Planned}} \times 100$$

Representativeness expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition or an environmental condition. Representativeness is a qualitative parameter which is dependent upon the proper design of the sampling program and proper laboratory protocol. The sampling network was designed to provide data representative of Site conditions. During development of this network, consideration was given to the historical Site operations, existing analytical data and physical setting and processes. The rationale of the sampling network is discussed in detail in the O&M Plan. Representativeness will be satisfied by insuring that the O&M Plan is followed, proper sampling techniques are used, proper analytical procedures are followed and holding times of the samples are not exceeded in the laboratory. Representativeness will be assessed by field duplicate sample data.

Comparability expresses the confidence with which one data set can be compared with another. The extent to which existing and planned analytical data will be comparable depends on the similarity of sampling and analytical methods. The procedures used to obtain the planned analytical data, as documented in the QAPP, are expected to provide

comparable data. These new analytical data, however, may not be directly comparable to existing data because of difference in procedures and QA objectives.

12.3.4 Field Measurements

Measurement data will be generated in many field activities. These activities include, but are not limited to, the following:

i) documenting time and weather conditions;
ii) determining pH, specific conductivity, and temperature of groundwater samples; and
iii) verifying pre-sampling purge volumes.

The general QA objective for such measurement data is to obtain reproducible and comparable measurements to a degree of accuracy consistent with the SOPs in Appendix 12.1.

12.8.2.4 MS/MSD, LS/LSD and MS/DUP Samples

A MS/MSD and LS/LSD sample set will be analyzed at a minimum frequency of one per twenty investigative samples for organic and air analyses, respectively. A MS/DUP or MS/MSD sample set will be analyzed for inorganic analyses at the same frequency as MS/MSD samples. Acceptance criteria and compounds that will be used for matrix spikes are identified in the SOPs in Appendix 12.1. Percent spike recoveries will be used to evaluate analytical accuracy while relative percent difference between the spike and matrix spike duplicate will be used to assess analytical precision.

12.8.2.5 Surrogates

Surrogates are used in all GC and GC/MS analyses. Every blank, standard, and environmental sample including MS/MSD samples will be spiked with surrogate compounds prior to purging volatiles or extracting semi-volatiles.

Surrogates will be spiked into samples according to the appropriate analytical methods. Surrogate spike recoveries will fall within the control limits set by procedures specified in the method for analytes falling within the quantitation limits without dilution. Dilution of samples to bring the analyte concentration into the linear range of calibration may dilute the surrogates out of the quantitation limit; assessment of analytical quality in these cases will be based in the quality control embodied in the check, matrix spike and matrix spike duplicate samples. Surrogate compounds recovery control limits will be those presented in the SOPs in Appendix 12.1.

12.9 DATA REDUCTION, VALIDATION AND REPORTING

The project laboratory will perform analytical data reduction and review in-house under the direction of the laboratory QA Officer. The laboratory QA Officer will be responsible for assessing data quality and advising of any data which were rated "preliminary" or "unacceptable" or other qualifications based on the established QC criteria. The laboratory will provide Level III (or equivalent) deliverables. Data reduction, review and reporting by the laboratory is typically conducted as detailed in the following procedure.

1. Raw data produced and checked by the responsible analyst is turned over for independent review by another analyst.

2. The area supervisor reviews the data for attainment of quality control criteria established by the QAPP.

3. The area supervisor will decide whether any sample re-analysis is required.

4. Upon completion of all reviews and acceptance of the raw data by the supervisor, a report will be generated and sent to the Project Manager.

5. The Project Manager will complete a thorough inspection of all reports.

6. Upon acceptance of the preliminary reports by the Project Manager, final reports will be generated and signed by the laboratory Operations Manager or his designee.

7. A thorough review of a percentage of all data packages is performed by the laboratory Quality Assurance Officer or his designee.

Field data from direct-reading instruments (pH, conductance, temperature) will not require reduction. Laboratory data reduction will be performed using the equations in the SOPs provided in Appendix 12.1.

CRA's QA/QC Officer - Analytical and Field Activities will conduct an evaluation of data reduction and reporting by the laboratory. These evaluations will consider the finished data sheets, field blank data and recovery data for surrogate and matrix spikes. The material will be checked for legibility, completeness, correctness and the presence of requisite dates, initials, and signatures. The results of these checks will be assessed and reported to the Engineering Consultant's Project Manager noting any discrepancies and their effect upon the acceptability of the data. All information garnered for QA/QC checks will be discussed in a QA/QC Validation report.

Validation of the analytical data will be performed by CRA's QA/QC Officer - Analytical and Field Activities based on the applicable evaluation criteria outlined in "USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review", EPA-540/R-94-012 and "USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review", EPA-540/R-94-013. The assessment of analytical and field data will include checks for adherence to laboratory QA procedures and accuracy and precision criteria; and the presence of transmittal errors and anomalously high or low parameter values. The results of these data validations will be reported to the Project Manager, noting any problems and their effect upon the acceptability of the data.

Data produced from field measurements and sample collection activities that are used in the project reports will be appropriately identified and appended to the report. Where data have been reduced or summarized, the method of reduction will be documented in the report. In

addition, field data will be audited for anomalously high or low values that may appear to be inconsistent with other data.

Laboratory data packages for chemical analyses will consist of the following deliverables:

i) a case narrative that includes a summary of analytical methods used and a description of any unusual action or conditions;

ii) dates of sample receipt, extraction/digestion and analysis;

iii) laboratory and field sample identification numbers;

iv) samples results in tabular format;

v) method blank sample summaries;

vi) surrogate compound recovery data and control limits;

vii) MS/MSD, LS/LSD and MS/DUP recovery and RPD data and control limits;

viii) check sample data; and

ix) executed chain-of-custody forms.

The data packages will be stored with the evidentiary files as described in Section 12.5.4. The USEPA and OEPA, upon request, will receive (within 30 days of receipt) all raw data packages from the project laboratories.

12.12 SPECIFIC ROUTINE PROCEDURES USED TO ASSESS DATA PRECISION, ACCURACY AND COMPLETENESS

The following sections include the procedures and formulae utilized to assess the levels of precision, accuracy and completeness achieved during the associated sample analyses.

12.12.1 Field Measurements

Field data will be assessed by the QA/QC Officer Analytical and Field Activities who will review the field results for compliance with the established QC criteria that are specified in the QAPP. Accuracy of the field measurements will be assessed using daily instrument calibration, calibration check, and analysis of blanks. Precision will be assessed on the basis of the reproducibility of duplicate readings of a single sample. Data completeness will be calculated using the following equation:

$$\text{Completeness (\%)} = \frac{\text{Valid (Usable) Data Obtained}}{\text{Total Data Planned}} \times 100$$

The required level of completeness will be 90 percent or greater.

12.12.2 Laboratory Data

Laboratory results will be assessed for compliance with required precision, accuracy, completeness and sensitivity as follows:

12.12.2.1 Precision

Precision of laboratory analysis will be assessed by comparing the analytical results between MS/MSD for organic analysis, LS/LSD for air analysis and MS/MSD or laboratory duplicate analyses for inorganic analysis. The relative percent difference (RPD) will be calculated for each pair of duplicate analyses as discussed in Section 12.12.3.

12.12.2.2 Accuracy

Accuracy of laboratory results will be assessed for compliance with the established QC criteria that are described in Sections 12.3 and 12.8 of the QAPP using the analytical results of method blanks, reagent/preparation blank, MS/MSD samples, LS/LSD samples, field blank and trip blanks. The percent recovery (%R) of matrix spike samples will be calculated as discussed in Section 12.12.3.

12.12.2.3 Completeness

Completeness will be assessed by comparing the number of usable results to the total possible number of results using the formula presented in Section 12.12.1. The required level of completeness for laboratory analyses will be 90 percent or greater.

12.12.2.4 Sensitivity

The achievement of targeted quantitation limits depend on instrumental sensitivity and matrix effects. Therefore, it is important to monitor the instrumental sensitivity to ensure the data quality through constant instrument performance. The instrumental sensitivity will be monitored through the analysis of method blank and calibration check standards.

12.12.3 Statistical Evaluations

In examination of data and determination of its precision and accuracy, standard statistical formulae will be used.

12.12.3.1 Arithmetic Mean

The arithmetic mean is the average obtained by dividing a sum by the number of its addends. A number of recovery results are averaged together to improve the accuracy of the measurement. Figure 12.3, equation 1 summarizes the formula to be used to determine the arithmetic mean.

12.12.3.2 Standard Deviation

The standard deviation is the square root of the average squared difference between the individual values and the average value. A number of recovery results are evaluated to find the numerical variation in the data which is then used in the determination of the percent relative standard deviation. Figure 12.3 equation 2 summarizes the formula to be used to determine the standard deviation.

12.12.3.3 Percent Relative Standard Deviation (%RSD)

The percent relative standard deviation is obtained by dividing the standard deviation of the values by the arithmetic mean of the values. The %RSD is calculated on a series of measurements to evaluate an

instruments analytical precision (e.g., initial calibration). Figure 12.3, equation 3 summarizes the formula to be used to determine %RSD.

12.12.3.4 Percent Recovery (%R)

The percent recovery of a parameter is obtained by dividing the amount recovered by the true amount added and multiplying by 100. The percent recoveries of spiked samples are evaluated to establish the analytical accuracy of a measurement. Figure 12.3, equation 4 summarizes the formula to be used to determine the percent recovery.

12.12.3.5 Relative Percent Difference (RPD)

The relative percent difference is obtained by dividing the difference between two numbers by their arithmetic mean and multiplying by 100. The RPD is used to evaluate the analytical precision of two replicate measurements (e.g., matrix spike/matrix spike duplicate). Figure 12.3, equation 5 summarizes the formula to be used to determine RPD.

TABLE 12.6

TARGETED QUANTITATION LIMITS FOR AIR ANALYSES
SUMMIT NATIONAL SUPERFUND SITE
DEERFIELD TOWNSHIP OF PORTAGE COUNTY, OHIO

Volatile Organic Compounds	*Targeted Quantitation Limits* [1] *Air (ppbv)* [2]
benzene	2.0
bromomethane	2.0
carbon tetrachloride	2.0
chlorobenzene	2.0
chloroethane	4.0
chloroform	2.0
chloromethane	4.0
1,3-dichloropropene	2.0
1,1-dichloroethane	2.0
1,2-dichloroethane	2.0
1,1-dichloroethene	2.0
1,2-dichloroethene	2.0
1,2-dichloropropane	2.0
ethylbenzene	2.0
methylene chloride	2.0
1,1,2,2-tetrachloroethane	2.0
tetrachloroethene	2.0
toluene	2.0
1,1,1-trichloroethane	2.0
1,1,2-trichloroethane	2.0
trichloroethene	2.0
vinyl chloride	2.0

[1] Actual sample quantitation limits are highly matrix and laboratory dependant and are not always achievable. Targeted quantitation limits presented are for guidance only and may not be achievable.

[2] ppbv = parts per billion by volume

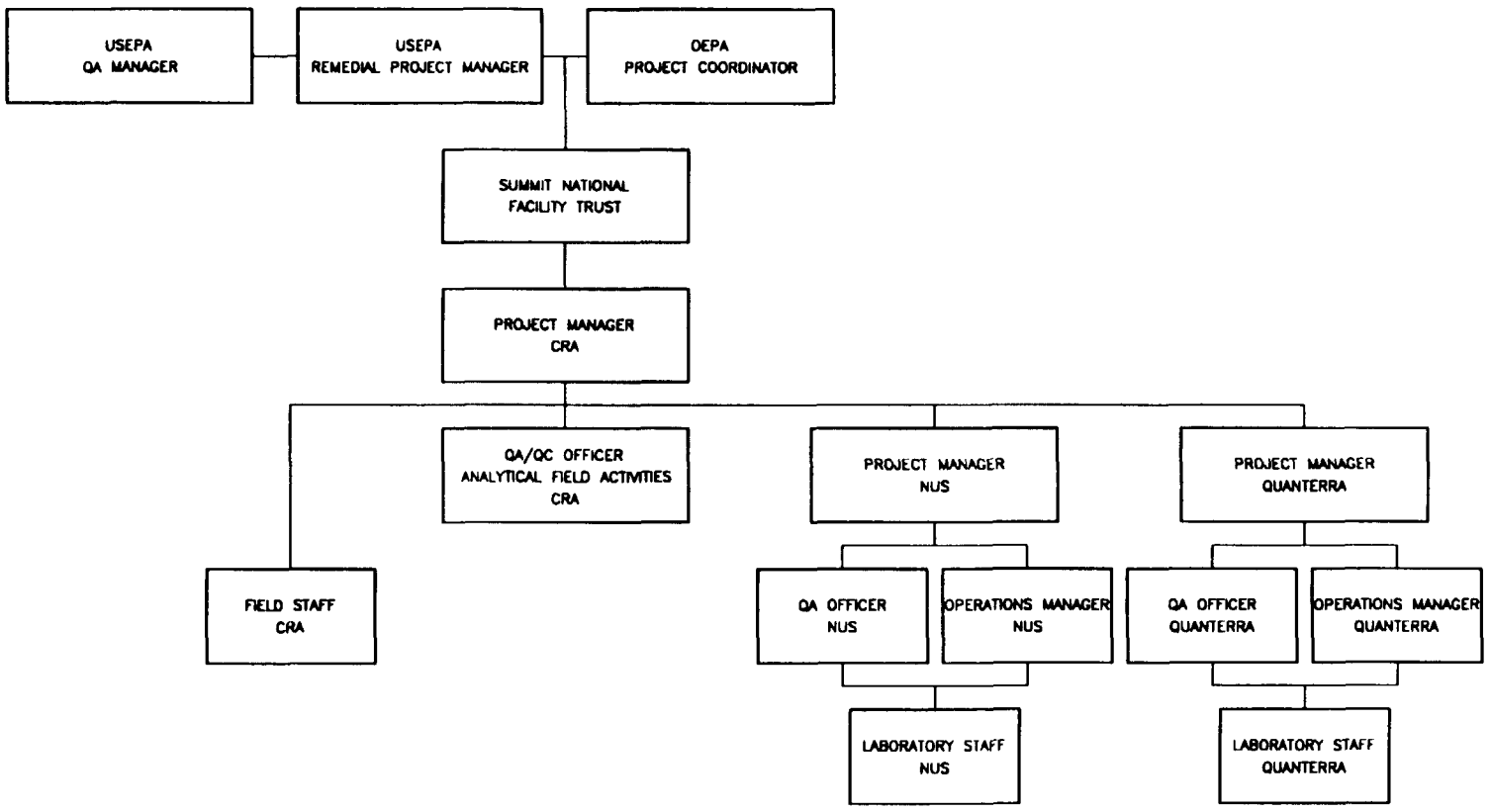

figure 12.1
QA/QC ORGANIZATION
SUMMIT NATIONAL SUPERFUND SITE
Deerfield Township Of Portage County, Ohio

CRA

Terri Wynnik - Sample Custodian - NUS

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6. Upon acceptance of the preliminary reports by the Project Manager, final reports will be generated and signed by the laboratory Operations Manager or his designee.

7. A thorough review of a percentage of all data packages is performed by the laboratory Quality Assurance Officer or his designee.

Field data from direct-reading instruments (pH, conductance, temperature) will not require reduction. Laboratory data reduction will be performed using the equations in the SOPs provided in Appendix 12.1.

CRA's QA/QC Officer - Analytical and Field Activities will conduct an evaluation of data reduction and reporting by the laboratory. These evaluations will consider the finished data sheets, field blank data and recovery data for surrogate and matrix spikes. The material will be checked for legibility, completeness, correctness and the presence of requisite dates, initials, and signatures. The results of these checks will be assessed and reported to the Engineering Consultant's Project Manager noting any discrepancies and their effect upon the acceptability of the data. All information garnered for QA/QC checks will be discussed in a QA/QC Validation report.

Validation of the analytical data will be performed by CRA's QA/QC Officer - Analytical and Field Activities based on the applicable evaluation criteria outlined in "USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review", EPA-540/R-94-012 and "USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review", EPA-540/R-94-013. The assessment of analytical and field data will include checks for adherence to laboratory QA procedures and accuracy and precision criteria; and the presence of transmittal errors and anomalously high or low parameter values. The results of these data validations will be reported to the Project Manager, noting any problems and their effect upon the acceptability of the data.

Data produced from field measurements and sample collection activities that are used in the project reports will be appropriately identified and appended to the report. Where data have been reduced or summarized, the method of reduction will be documented in the report. In

addition, field data will be audited for anomalously high or low values that may appear to be inconsistent with other data.

Laboratory data packages for chemical analyses will consist of the following deliverables:

i) a case narrative that includes a summary of analytical methods used and a description of any unusual action or conditions;

ii) dates of sample receipt, extraction/digestion and analysis;

iii) laboratory and field sample identification numbers;

iv) samples results in tabular format;

v) method blank sample summaries;

vi) surrogate compound recovery data and control limits;

vii) MS/MSD, LS/LSD and MS/DUP recovery and RPD data and control limits;

viii) check sample data; and

ix) executed chain-of-custody forms.

The data packages will be stored with the evidentiary files as described in Section 12.5.4. The USEPA and OEPA, upon request, will receive (within 30 days of receipt) all raw data packages from the project laboratories.

12.12 SPECIFIC ROUTINE PROCEDURES USED TO ASSESS DATA PRECISION, ACCURACY AND COMPLETENESS

The following sections include the procedures and formulae utilized to assess the levels of precision, accuracy and completeness achieved during the associated sample analyses.

12.12.1 Field Measurements

Field data will be assessed by the QA/QC Officer Analytical and Field Activities who will review the field results for compliance with the established QC criteria that are specified in the QAPP. Accuracy of the field measurements will be assessed using daily instrument calibration, calibration check, and analysis of blanks. Precision will be assessed on the basis of the reproducibility of duplicate readings of a single sample. Data completeness will be calculated using the following equation:

$$\text{Completeness (\%)} = \frac{\text{Valid (Usable) Data Obtained}}{\text{Total Data Planned}} \text{ X } 100$$

The required level of completeness will be 90 percent or greater.

12.12.2 Laboratory Data

Laboratory results will be assessed for compliance with required precision, accuracy, completeness and sensitivity as follows:

12.12.2.1 Precision

Precision of laboratory analysis will be assessed by comparing the analytical results between MS/MSD for organic analysis, LS/LSD for air analysis and MS/MSD or laboratory duplicate analyses for inorganic analysis. The relative percent difference (RPD) will be calculated for each pair of duplicate analyses as discussed in Section 12.12.3.

12.12.2.2 Accuracy

Accuracy of laboratory results will be assessed for compliance with the established QC criteria that are described in Sections 12.3 and 12.8 of the QAPP using the analytical results of method blanks, reagent/preparation blank, MS/MSD samples, LS/LSD samples, field blank and trip blanks. The percent recovery (%R) of matrix spike samples will be calculated as discussed in Section 12.12.3.

12.12.2.3 Completeness

Completeness will be assessed by comparing the number of usable results to the total possible number of results using the formula presented in Section 12.12.1. The required level of completeness for laboratory analyses will be 90 percent or greater.

12.12.2.4 Sensitivity

The achievement of targeted quantitation limits depend on instrumental sensitivity and matrix effects. Therefore, it is important to monitor the instrumental sensitivity to ensure the data quality through constant instrument performance. The instrumental sensitivity will be monitored through the analysis of method blank and calibration check standards.

12.12.3 Statistical Evaluations

In examination of data and determination of its precision and accuracy, standard statistical formulae will be used.

12.12.3.1 Arithmetic Mean

The arithmetic mean is the average obtained by dividing a sum by the number of its addends. A number of recovery results are averaged together to improve the accuracy of the measurement. Figure 12.3, equation 1 summarizes the formula to be used to determine the arithmetic mean.

12.12.3.2 Standard Deviation

The standard deviation is the square root of the average squared difference between the individual values and the average value. A number of recovery results are evaluated to find the numerical variation in the data which is then used in the determination of the percent relative standard deviation. Figure 12.3 equation 2 summarizes the formula to be used to determine the standard deviation.

12.12.3.3 Percent Relative Standard Deviation (%RSD)

The percent relative standard deviation is obtained by dividing the standard deviation of the values by the arithmetic mean of the values. The %RSD is calculated on a series of measurements to evaluate an

instruments analytical precision (e.g., initial calibration). Figure 12.3, equation 3 summarizes the formula to be used to determine %RSD.

12.12.3.4 Percent Recovery (%R)

The percent recovery of a parameter is obtained by dividing the amount recovered by the true amount added and multiplying by 100. The percent recoveries of spiked samples are evaluated to establish the analytical accuracy of a measurement. Figure 12.3, equation 4 summarizes the formula to be used to determine the percent recovery.

12.12.3.5 Relative Percent Difference (RPD)

The relative percent difference is obtained by dividing the difference between two numbers by their arithmetic mean and multiplying by 100. The RPD is used to evaluate the analytical precision of two replicate measurements (e.g., matrix spike/matrix spike duplicate). Figure 12.3, equation 5 summarizes the formula to be used to determine RPD.

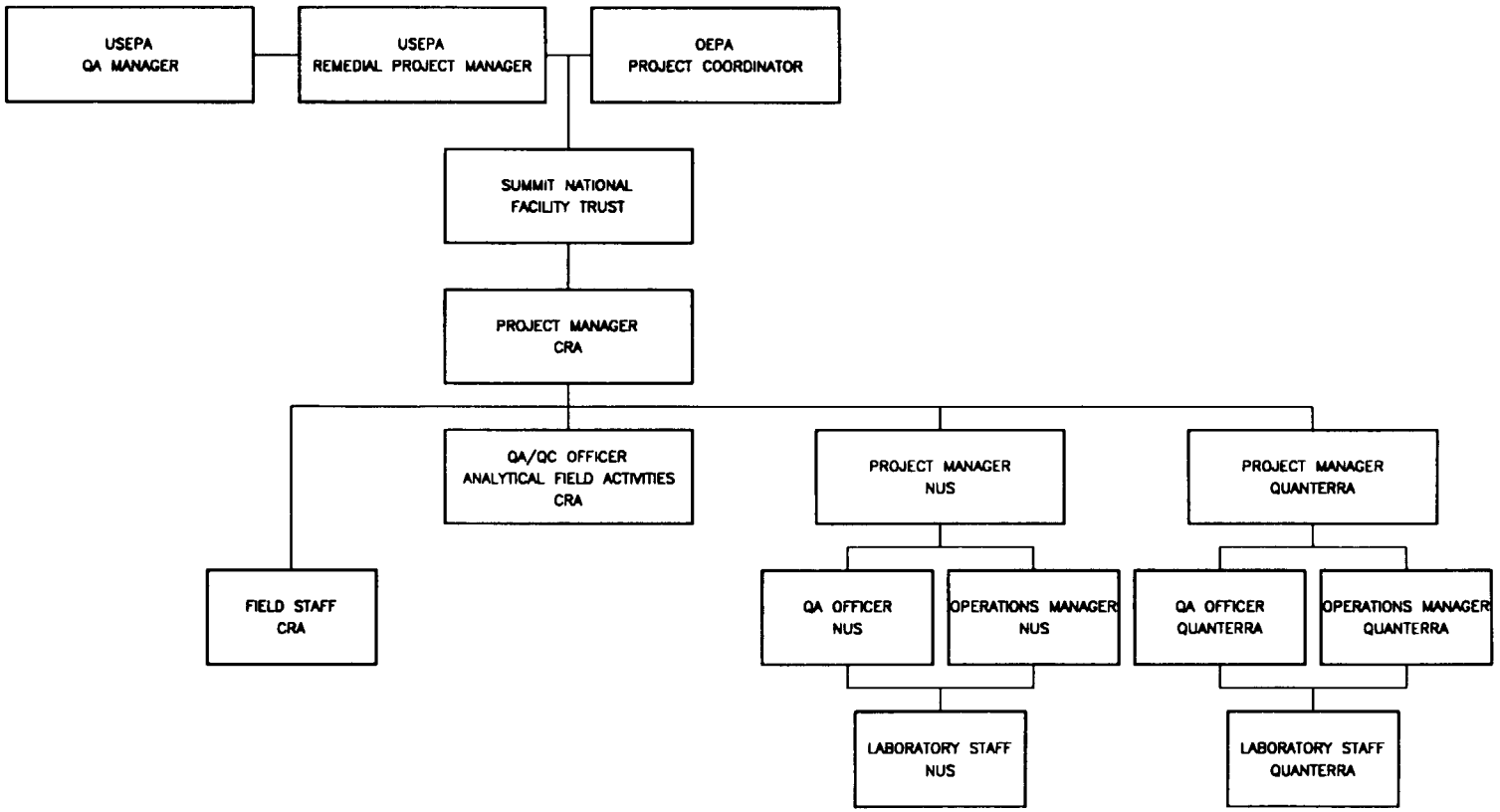

figure 12.1
QA/QC ORGANIZATION
SUMMIT NATIONAL SUPERFUND SITE
Deerfield Township Of Portage County, Ohio

CRA

TABLE 12.6

TARGETED QUANTITATION LIMITS FOR AIR ANALYSES
SUMMIT NATIONAL SUPERFUND SITE
DEERFIELD TOWNSHIP OF PORTAGE COUNTY, OHIO

	Targeted Quantitation Limits [1] *Air (ppbv)* [2]
Volatile Organic Compounds	
benzene	2.0
bromomethane	2.0
carbon tetrachloride	2.0
chlorobenzene	2.0
chloroethane	4.0
chloroform	2.0
chloromethane	4.0
1,3-dichloropropene	2.0
1,1-dichloroethane	2.0
1,2-dichloroethane	2.0
1,1-dichloroethene	2.0
1,2-dichloroethene	2.0
1,2-dichloropropane	2.0
ethylbenzene	2.0
methylene chloride	2.0
1,1,2,2-tetrachloroethane	2.0
tetrachloroethene	2.0
toluene	2.0
1,1,1-trichloroethane	2.0
1,1,2-trichloroethane	2.0
trichloroethene	2.0
vinyl chloride	2.0

[1] Actual sample quantitation limits are highly matrix and laboratory dependant and are not always achievable. Targeted quantitation limits presented are for guidance only and may not be achievable.

[2] ppbv = parts per billion by volume

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STANDARD
OPERATING
PROCEDURE

Subject or Title: **EPA Method TO-14** Page 1 of 39
The Determination of Volatile Organics (VOCs)
in Ambient Air by GC/MS - Scan Mode

SOP No.:	Revision No.: 2.3	Effective Date:
LM-ATL-7001	Supersedes: rev. 2.2	July 1, 1993

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Prepared by: Steve Harris **Date:** 6/28/93
Revised by: Joe Konschnik/Mark Johnson

Management Approval: **Date:** 7/11/93

QA Officer Approval: **Date:** 9-1-93

Quanterra
Environmental Services

STANDARD OPERATING PROCEDURE

Subject or Title:	**EPA Method TO-14**	Page 1 of 29
	The Determination of Volatile Organics (VOCs) in Ambient Air by GC/MS - Scan Mode	
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Prepared by: Steve Harris Revised by: Joe Konschnik/Mark Johnson	Date: 6/28/93
Management Approval:	Date:
QA Officer Approval:	Date:

1. Scope and Application

1.1 Analytes (See Table 1)

1.2 Method detection limits (See Table 1)

1.3 Reporting limits (See Table 12)

1.4 Applicable matrices - air, vapor

1.5 Dynamic range (See Table 1)

1.6 Approximate analytical time

2 min. - cool down of cryotrap
2 min. - flush of inlet system with internal standard
2 min. - collection of 100 mL internal standard on trap
2 min. - flush of inlet system on trap
10 min. - collection of 500 mL of sample/standard on trap
2 min. - flush of trap with HP Helium and GC oven cool down
24 min. - GC run time

When running multiple samples, steps can be overlapped to reduce run time to approximately 30 min.

TABLE 1. VOC Target Compounds

	Compound	R.T.	Detection Limits MDL (ppbv)	Dynamic Range (ppbv)
2)	Dichlorodifluoromethane (Freon 12)	2.05	0.066	2-450
3)	Chloromethane	2.62	0.086	4-450
4)	1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon 114)	2.66	0.046	2-290
5)	Vinyl chloride	2.89	0.20	2-450
6)	Bromomethane	3.34	0.072	2-450
7)	Chloroethane	3.52	0.12	4-450
8)	Trichlorofluoromethane (11)	3.85	0.046	2-290
9)	1,1-Dichloroethene	4.44	0.035	2-450
10)	Carbon disulfide	4.54	0.049	2-360
11)	1,1,2-Trichloro- 1,2,2-trifluoroethane (Freon 113)	4.52	0.038	2-290
12)	Acetone	4.62	0.063	10-450
13)	Methylene chloride	5.07	0.043	2-450
14)	trans-1,2-Dichloroethene	5.38	0.040	2-450
15)	1,1-Dichloroethane	5.91	0.028	2-450
16)	Vinyl Acetate	6.13	0.10	10-450
17)	cis-1,2-Dichloroethene	6.70	0.045	2-450
18)	2-Butanone	6.84	0.12	5-450
19)	Chloroform	7.23	0.033	2-450
20)	1,1,1-Trichloroethane	7.33	0.016	2-450
21)	Carbon tetrachloride	7.56	0.021	2-290
23)	Benzene	7.88	0.063	2-450
24)	1,2-Dichloroethane	7.97	0.032	2-450
25)	Trichloroethene	8.95	0.014	2-450
26)	1,2-Dichloropropane	9.31	0.033	2-450
27)	Bromodichloromethane	9.88	0.056	2-450
28)	cis-1,3-Dichloropropene	10.70	0.019	2-450
29)	4-Methyl-2-pentanone	11.11	0.085	4-450
30)	Toluene	11.21	0.059	2-450
32)	trans-1,3-Dichloropropene	11.82	0.085	2-450
33)	1,1,2-Trichloroethane	12.12	0.084	2-450
34)	Tetrachloroethene	12.19	0.055	2-450
35)	2-Hexanone	12.78	0.12	4-450
36)	Dibromochloromethane	12.80	0.048	2-450
37)	1,2-Dibromoethane	12.90	0.031	2-450
38)	Chlorobenzene	13.98	0.026	2-450
39)	Ethylbenzene	14.33	0.035	2-450
40)	1,4-and 1,3-(p,m) Xylene	14.61	0.14	2-580

TABLE 1. VOC Target Compounds (Continued)

	Compound	R.T.	Detection Limits MDL (ppbv)	Dynamic Range
41)	1,2-(ortho) Xylene	15.43	0.14	2-450
42)	Styrene	15.49	0.083	2-450
43)	Bromoform	15.78	0.17	2-450
44)	1,1,2,2-Tetrachloroethane	17.18	0.055	2-450
45)	Benzyl chloride	17.30	0.029	2-450
46)	4-Ethyltoluene	17.55	0.079	2-450
47)	1,3,5-Trimethylbenzene	17.73	0.072	2-450
48)	1,2,4-Trimethylbenzene	18.55	0.064	2-450
49)	1,3-Dichlorobenzene	19.02	0.075	2-450
50)	1,4-Dichlorobenzene	19.26	0.11	2-450
51)	1,2-Dichlorobenzene	19.83	0.087	2-450
52)	1,2,4-Trichlorobenzene	21.32	0.16	4-450
53)	Hexachlorobutadiene	21.52	0.071	4-450

2. Summary of Method

2.1 A pressurized air sample is metered through a mass flow controller onto a cryogenically cooled trap. After 100 mL of internal standard and 500 mL of the sample has been trapped, a valve is switched and the trap is heated to purge the trap's contents onto the gas chromatography column. The target compounds are analyzed with a mass spectrometer operated in the scan mode.

3. Comments

3.1 Interferences

3.1.1 Gas regulators are cleaned by the manufacturer using Freon 113, which is one of the target compounds. Before using ultra high purity (UHP) Nitrogen (N_2), Hydrocarbon (HC) free air, Internal Standard (I.S.), or a target compound standard mix, each regulator should be purged a minimum of three times with the appropriate gas.

3.1.2 Contamination may occur in the sampling system if canisters are not properly cleaned prior to use. Canisters should not be used for the collection of samples until a batch blank analysis indicates that no target compounds are present above 0.2 ppbv. All other sampling equipment including pumps, flow controllers and filters must be thoroughly cleaned to ensure that the filling apparatus will not contaminate samples.

3.1.3 High levels of CO2 and/or moisture may limit the amount of sample that can be trapped due to plugging of the trap. High levels of CO2 may also over-pressurize the instrument's vacuum system requiring delay of scan start time.

3.2 Helpful Hints

None

4. Safety Issues

4.1 In order to prevent contamination of the lab air by the samples, the vent line must be connected to the system outlet and the fume hood must be on.

4.2 While making standards, the fume hood must be running. When finished valves must be closed and lines vented.

4.3 All compressed gas cylinders must be securely fastened to a bench or wall.

4.4 Normal precautions should be used in the handling of liquid nitrogen (LN_2) (do not touch transfer lines as burns can result).

4.5 Sampling canisters should never be pressurized over 40 psig.

5. Sample Collection, Preservation, Containers and Holding Times

5.1 Samples should be collected in precleaned and batch analyzed SUMMA passivated canisters. A 7 micron filter should be placed on the inlet of the can to protect the valve from particulates. Canisters should never be pressurized over 40 psig.

5.2 The absolute pressure of the canister must be recorded before and after sample collection.

5.3 Samples must be kept at <25°C.

5.4 Canister samples should be analyzed within 14 days of collection.

6. Apparatus and Materials

6.1 Gas chromatograph - capable of subambient temperature programming for the oven and with the jet separator option (Hewlett Packard 5890).

6.2 Mass-selective detector - equipped with computer and appropriate software (Hewlett Packard 5970B with HP-1000 RTE-A data system).

6.3 Cryogenic trap with temperature control assembly (Nutech 8533 and 3538). See Figure 1.

6.4 Electronic mass flow controller - for maintaining constant sample flow through Nutech concentrators (Unit Instruments)

6.5 Chromatographic grade stainless steel tubing and stainless steel plumbing fittings.

6.6 Chromatographic column - DB-624 0.53 ID, 30 meter length (J&W Scientific).

6.7 Stainless steel vacuum/pressure gauge capable of measuring from 30" of mercury (Hg) to 40 psig. (Span Instruments)

6.8 High precision vacuum gauge - for making daily standards. (Wallace & Tiernan Pennwalt)

6.9 Pressure regulators for carrier gas and standards - 2 stage, stainless steel diaphragm.

6.10 SUMMA passivated canisters 6 L or 15 L (Scientific Instrumentation Specialists, Anderson Instruments) or equivalent.

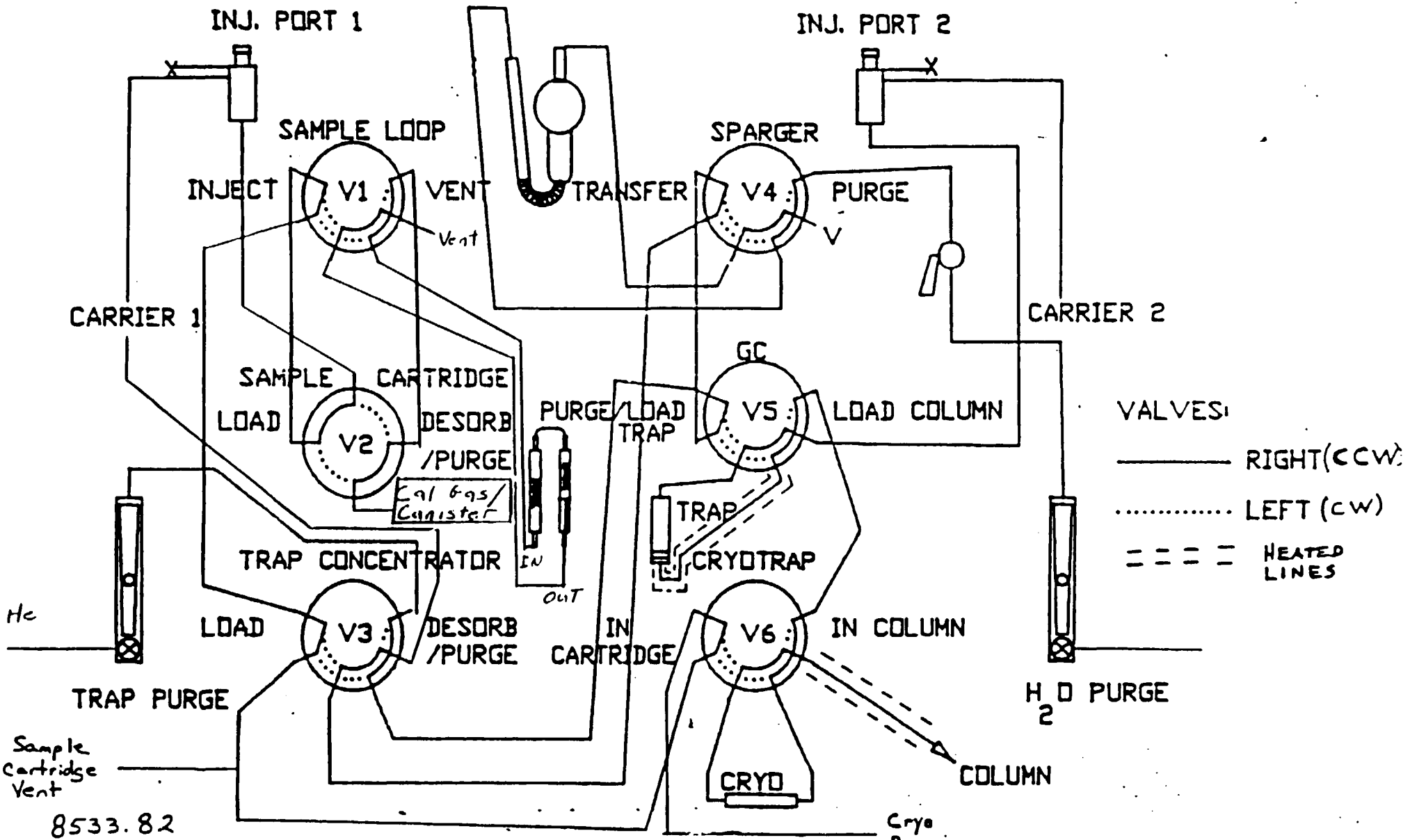
6.11 7 micron filters (Nupro), or equivalent.

FIGURE 1. Nutech 8533 Flow Diagram

8/9, 10

8/9/90

Ensco Revised



8533.82

w/ Cryo

VOST ANALYZER FLOW DIAGRAM (FRONT VIEW)

7. Reagents and Standards

7.1 4-bromofluorobenzene, 25 ng/uL in methanol (for tuning of mass spectrometer).

7.2 High purity helium and air for making dilutions and for carrier gas.

7.3 Calibration stock standards are at a nominal concentration of 1 ppmv (CS_2 is not as stable and so the concentration is 5 ppmv). Standards are prepared in a balance gas of nitrogen and are analytically certified by the supplier (Scott-Marrin and Scott Specialty). To facilitate certification by vendor, the standards were divided into 5 cylinders. (See Tables 2-7.)

7.4 Internal standard mix of bromochloromethane, 1,4-difluorobenzene, and chlorobenzene-d5 at 1000 ug/ml each in methanol (Supelco).

TABLE 2. Cylinder No. CC72069

Component	Concentration (v/v)
Chloromethane	0.98 ± 0.05 ppmv
Bromomethane	1.00 ± 0.05 ppmv
Chloroethane	0.96 ± 0.05 ppmv
Dichloromethane	1.08 ± 0.05 ppmv
trans-1,2-Dichloroethylene	1.08 ± 0.05 ppmv
Trichloroethane	1.07 ± 0.05 ppmv
1,2-Dichloroethane	1.10 ± 0.05 ppmv
1,1,1-Trichloroethane	0.99 ± 0.05 ppmv
Tetrachloromethane	1.01 ± 0.05 ppmv
1,2-Dichloropropane	1.08 ± 0.05 ppmv
cis-1,3-Dichloropropene	1.03 ± 0.05 ppmv
trans-1,3-dichloropropene	1.20 ± 0.06 ppmv
Dibromochloromethane	1.13 ± 0.05 ppmv
Tetrachloroethylene	1.14 ± 0.05 ppmv
Ethylbenzene	1.20 ± 0.06 ppmv
p-Xylene	1.20 ± 0.06 ppmv
Styrene	1.25 ± 0.06 ppmv
1,1,2,2-Tetrachloroethane	1.24 ± 0.06 ppmv
Bromodichloromethane	1.08 ± 0.05 ppmv
Trichloroethene	0.82 ± 0.05 ppmv
Acetonitrile	1.00 ± 0.05 ppmv
Nitrogen	Balance

TABLE 3. Standard Cylinder No. CC72058

Component	Concentration (v/v)
Carbon Disulfide	4.86 ± 0.1 ppmv
Nitrogen	Balance

TABLE 4. Standard Cylinder No. CC72063

Component	Concentration (v/v)
Vinyl Chloride	1.00 ± 0.05 ppmv
1,1-Dichloroethene	1.08 ± 0.05 ppmv
1,1-Dichloroethane	1.06 ± 0.05 ppmv
2-Butanone	1.02 ± 0.05 ppmv
cis-1,2-Dichloroethene	1.07 ± 0.05 ppmv
Benzene	1.07 ± 0.05 ppmv
4-Methyl-2-pentanone	1.09 ± 0.05 ppmv
1,1,2-Trichloroethane	1.06 ± 0.05 ppmv
Toluene	1.08 ± 0.05 ppmv
2-Hexanone	1.18 ± 0.05 ppmv
Chlorobenzene	1.08 ± 0.05 ppmv
m-Xylene	1.11 ± 0.05 ppmv
o-Xylene	1.12 ± 0.05 ppmv
1,2-Dichlorobenzene	1.25 ± 0.05 ppmv
Acetone	0.99 ± 0.05 ppmv
1,4-Dichlorobenzene	1.04 ± 0.05 ppmv
Nitrogen	Balance

TABLE 5. Standard Cylinder No. CC12390

Component	Concentration (v/v)
Freon-12	1.015 ± 0.05 ppmv
Freon-114	0.95 ± 0.05 ppmv
Freon-11	0.94 ± 0.05 ppmv
Freon-113	0.99 ± 0.05 ppmv
n-Hexane	1.02 ± 0.05 ppmv
1,2-Dibromoethane	0.99 ± 0.05 ppmv
4-Ethyltoluene	0.89 ± 0.05 ppmv
1,3,5-Trimethylbenzene	0.95 ± 0.05 ppmv
1,2,4-Trimethylbenzene	0.92 ± 0.05 ppmv
Nitrogen	Balance

TABLE 6. Internal Standard Liquid Mix

Component	Concentration (ug/ml)
Bromochloromethane	1000
1,4-D,fluorobenzene	1000
Chlorobenzene-d5	1000

TABLE 7. Standard Cylinder No. ALM 002636

Component	Concentration (v/v)
Benzyl chloride	0.737 ppmv
1,3-Dichlorobenzene	0.768 ppmv
1,4-Dioxane	0.895 ppmv
Hexachloro-1,3-butadiene	0.804 ppmv
Bromoform	0.84 ppmv
1,2,4-Trichlorobenzene	0.898 ppmv
Vinyl acetate	0.838 ppmv
Nitrogen	Balance

8. Procedure

8.1 Sample Preparation

8.1.1 The pressure of the sample canister is checked and recorded by attaching a vacuum/pressure gauge to the top valve of the canister (the gauge should be rinsed for few seconds with HC free air by physically holding against the air outlet and flushing). The canister valve is opened briefly and the pressure is recorded. If the pressure is less than 10 psig, pressurize the canister to 10 psig with HC free air.

8.1.2 If the canister pressure is increased, a dilution factor (DF) is calculated and recorded.

$$DF = \frac{Y_a}{X_a}$$

Where: X_a = absolute canister pressure before dilution

Y_a = absolute canister pressure after dilution

8.2 Daily GC/MS Tuning

8.2.1 At the beginning of each day, or 12 hour shift, prior to a calibration, the GC/MS system must be tuned to verify that acceptable performance criteria are achieved. If any of the key ions fail the abundance criteria listed in Table 8, the system must be retuned using 4-Bromofluorobenzene (BFB).

8.2.2 For tuning, the cryotrap is not used and should be left at 150C. Alternatively the trap can be cold or cooling down but should be out of line with the column. The GC program (see Table 9) is initiated by using the BAMON commands. The GC programs are named "GCBFB1" and "GCBFB2." This downloads the program from the data system to the GC. Once the oven has stabilized, the remote start light will turn on and the system is ready for injection.

2 uL of a 25 ng/uL 4-bromofluorobenzene (BFB) standard is injected into the injection port of the Nutech and the remote start button is activated.

TABLE 8. 4-Bromofluorobenzene Key Ions and Ion Abundance Criteria

Mass	Ion Abundance Criteria
50	15 to 40% of mass 95
75	30 to 60% of mass 95
95	Base Peak, 100% Relative Abundance
96	5 to 9% of mass 95
173	<2% of mass 174
174	>50% of mass 95
175	5 to 9% of mass 174
176	>95% but <101% of mass 174
177	5 to 9% of mass 176

8.2.3 Once the tuning run is complete (~ 6 minutes), type in the command: "TRF, TUNVOA, data file". This will start a program that will evaluate the tuning analysis and print out the required information automatically. If the BFB tuning criteria cannot be met on the first injection, retuning the instrument with PFTBA and or system maintenance may be required prior to re-injection of BFB.

TABLE 9. BFB Tuning Method

Enter the name of the method file: GCBFB1

M E T H O D F I L E L I S T

Method file: GCBFB1
GCBFB2

GC type: 5890
Column: Cap

Run type: SCAN, GC, E1
Splitless: Yes

Temperature:	Inj.P	Intfc	Source
	90.0	250.0	0.0

GC/DIP		LEVEL A	LEVEL B	POST RUN
Temp 1	30.0	100.0	0.0	0.0
Time 1	1.0	3.0	0.0	0.0
Rate	35.0	0.0	0.0	
Temp 2	100.0	0.0	0.0	
Time 2	15.0	0.0	0.0	

Oven equilibration Time .10 min

Run time: 6.00
Scan Start time: 2.50

Scan Parameters: Mass Range 34 to 260
Multiplier voltage: varies Number of A/D samples: 8
GC Peak threshold: 20000 counts
Threshold: 100 counts

TABLE 10. Analytical Method

Enter the name of the method file: GCSYS1

M E T H O D F I L E L I S T

Method file: GCSYS1 GC type: 5890 Run type: SCAN, GC, E1
GCSYS2 Column: Cap Splitless: Yes

Temperature:	Inj.P	Intfc	Source
	90.0	250.0	0.0

GC/DIP		LEVEL A	LEVEL B	POST RUN
Temp 1	-50.0	10.0	100.0	0.0
Time 1	1.5	0.0	0.0	0.0
Rate	50.0	5.5	40.0	
Temp 2	10.0	100.0	160.0	
Time 2	0.0	0.0	3.4	

Oven equilibration Time .10 min

Run time: 24.00
Scan Start time: 1.60

Scan Parameters: Mass Range 34 to 260
Multiplier voltage: varies Number of A/D samples: 8
GC Peak threshold: 20000 counts
Threshold: 10 counts

8.3 Calibration

8.3.1 A static dilution of the stock standard gas mixtures is made in a 6 liter SUMMA canister. The high precision vacuum gauge is flushed with HC free air and attached to the top valve of a clean, evacuated canister. After recording the absolute pressure, 2.00 psi of each of the 4 standard mixtures and 0.50 psi of CS2 is added to the canister (each regulator and the transfer line must be flushed several times before transfer of standard to the canister). Close the canister valves and replace the high precision gauge with a vacuum/pressure gauge. Pressurize the can with HC free humid air to 30 psig. This will yield a standard with a nominal concentration of 45 ppbv for most compounds (see Table 11).

8.3.2 An initial, minimum 5 point, curve is run in the linear working range of the system for each compound. The nominal concentration of the 6 standards will be 2.2, 11, 44, 140, 290, and 450 ppbv. A relative standard deviation (RSD) is calculated for each target analyte using the calculation in section 11 and that analyte's dynamic range. 90% of the target compounds must be less than 30% RSD to accept the curve for analysis. Refer to section 11 for RSD caculation. All compounds should have a response factor greater than 0.05.

8.3.3 On a daily basis, or every 12 hours of operation, a one point midrange standard (500 ml of 44 ppbv) is run to verify linearity with the 5 point curve. A percent difference is calculated between the response factors(RFs) from the continuing calibration standard and the average RFs from the initial curve for each target analyte using the calculation in section 11. 90% of the target compounds response factors must be within 30% difference of the 5 point curve average Response Factor, or a new 5 point must be run. All compounds should have a response factor greater than 0.05. The daily, one point check standard response factors (RFs) are used to quantitate the results of the samples for that shift.

8.4 Analysis

8.4.1 The check standard and the QA/QC samples are analyzed the same as samples. After the one-point continuing calibration standard is analyzed and evaluated, duplicate laboratory control samples (DCS) are analyzed and evaluated (see sections 10.3 and 10.4).

8.4.2 Immediately following the duplicate control samples a hydrocarbon free air method blank is analyzed and evaluated (see section 10.5). The method blank consists of a SUMMA canister that is filled and pressurized with zero grade hydrocarbon free humid air.

8.4.3 At the beginning of a sample or standard run the sample valve is in the purge position and the trap valve is in the column position. The trap is cooled to -165C. The internal standard canister is attached and approximately 100mL is flushed through (this can be concurrent with the trap cool down). After flushing the trap valve is placed in the sample position and the trap temperature is checked to be at least -165C.

Trapping is started by switching the sample valve to sample position and activating a timer. When 100 mL has been trapped (2 minutes at 50mL/min) the sample valve is switched to purge and the internal standard canister is closed and removed.

8.4.4 The sample/standard canister is then attached and approximately 100mL is flushed through. Trapping is again started by switching the sample valve to sample and starting a timer. After the appropriate amount of time (10 minutes for 500mL at 50mL/min) the sample valve is switched back to purge, the canister is closed and the data system is set up.

8.4.5 The GC system methods are "GCSYS1" and "GCSYS2" (see Table 10). When the GC reaches ready at the programmed start temperature the trap valve is switched to column, the cryotrap begins heating and the run is started simultaneously.

8.4.6 A canister filled with zero grade air is attached to the sample line in between samples. The line is allowed to flush until it is time for the next sample.

9. Data Interpretation

9.1 Qualitative Analyses

9.1.1 An analyte (e.g., those listed in Table 1) is identified by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). Standard reference mass spectra are obtained on each of Enseco's GC/MS systems. These standard reference spectra may be obtained through analysis of the calibration standards. Two criteria must be satisfied to verify positive identification. (1) elution of sample component at the same GC relative or absolute retention time as those of the standard component; and (2) correspondence of the sample component and the standard component mass spectrum.

9.1.1.1 The sample component relative retention time (RRT) must compare within ± 0.06 RRT units of the RRT of the standard component. As an option, RT must compare within 0.5 minutes of the standard component absolute retention time (RT). For reference, the standard must be run within the same 12 hours as the sample.

9.1.1.2 (1) All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) must be present in the sample spectrum. (2) The relative intensities of ions specified in (1) must agree within plus or minus 20% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be must be between 30 and 70 percent.

9.1.1.3 If a compound cannot be verified by all of the criteria in the above paragraphs but in the technical judgement of the analytical chemist the identification is correct, then the compound may be reported.

9.1.2 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the type of analyses being conducted. Guidelines for making tentative identification are:

(1) Relative intensities of major ions in the reference spectrum (ions >10% of the most abundant ion) should be present in the sample spectrum.

(2) The relative intensities of the major ions should agree within ± 20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%).

(3) Molecular ions present in the reference spectrum should be present in the sample spectrum.

(4) Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

(5) Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.

(6) Only peaks having a total ion current greater than 10% of the nearest eluting Internal Standard total ion current will be evaluated for reporting.

(7) Semiquantitative results will be calculated for tentatively identified compounds using total ion current areas and assuming a relative response factor of 1.0.

Computer generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual comparison of sample with the nearest library searches will the mass spectral interpretation specialist assign a tentative identification.

9.2 Quantitative Analysis:

When a compound has been identified, the quantification of that compound will be based on the integrated abundance from the EICP of the primary charateristic ion. Quantification will take place using the internal standard technique. A summary table of internal standards and their corresponding primary and secondary ions is represented by Table 13. The calculation for quantitation of sample is found in section 11.

10. QA/QC Requirements

10.1 The mass spectrometer must meet the tuning criteria described in Section 8.2.

10.2 After tuning, a single point check standard must be analyzed. Ninety percent of the target compound response factors must be within ± 30 percent difference of the five point calibration curve average response factors. If the check standard fails to meet this criterion, the system conditions should be evaluated and the standard reanalyzed. If the reanalysis fails upon several attempts to meet linearity criteria a new five point calibration curve must be run.

10.3 A laboratory control sample (LCS) must be analyzed after the check standard. This sample will consist of the target VOCs prepared in a separate canister at a concentration that differs from that of the check standard. Five compounds will be used to assess control for the LCS: methylene chloride, 1,1-dichloroethene, trichloroethene, toluene and 1,1,2,2-tetrachloroethane. The percent recovery for the five control compounds must be within a window of 80-115% or a window established using historical lab data.

10.4 For each lot of 20 samples analyzed, a duplicate control sample (DCS) must be analyzed after the LCS. The DCS sample is identical to the LCS in composition and source. The same LCS percent recovery criterion must be met. In addition, the relative percent difference (RPD) for the LCS and DCS must be ≤ 20%. If either of the DCS fail the criteria the system should be checked and the LCS that failed reanalyzed. Samples will not be analyzed until the DCS criteria are met. QC sample limits may change once established using historical lab data.

10.5 A method blank must be analyzed after the LCS or DCS. The blank results must indicate that there are no target compounds present above the reporting limits (RL). The method blank is prepared by adding zero grade humid air to a SUMMA canister. Internal standards are added to the trap and the blank is processed exactly as a sample or standard.

10.6 If any of the above criteria are not met, corrective actions must be implemented before analyses can proceed.

10.7 Internal Standards and their associated key ions are noted in Table 13. The internal standard areas are monitored for each shift by comparing the areas of the internal standards in each sample with the areas of the internal standards in the daily continuing calibration standard. Sample areas are considered acceptable if they fall between 50% and 150% of the daily standard areas. Any sample exceeding this criterium should be documented either on the analysis benchsheet or in the report narrative. The internal standard area of bromochloromethane should always be greater than 110,000 area counts and less than 170,000 area counts.

11. Calculations

11.1 The HP data system automatically quantitates the sample results based on a 500 mL sample size. The results are in ppbv. If the canister was pressurized before analysis, the results must be multiplied by the dilution factor DF (see Section 8.1.2).

11.2 If a sample size other than 500 mL was used and/or a canister sample was pressurized, the result must be adjusted as shown below:

$$\text{result ppbv} \times \frac{500\ \text{mL}}{\text{sample volume injected}} \times \frac{\text{final psia}}{\text{initial psia}}$$

11.3 Calculation for Relative Response Factor (RRF):

$$RRF = \frac{\text{Area cpd in Std.}}{\text{Area I.S.}} \times \frac{\text{Conc. I.S.}}{\text{Conc. Cpd. in Std.}}$$

The area of the primary quantitation ion is used in calculation.
I.S.: Internal Standard

11.4 Calculation for Percent Relative Standard Deviation (%RSD):

$$\%RSD = \frac{\text{Std. Dev. of RRFs}}{\text{Mean of RRFs}} \times 100$$

11.5 Calculation for Percent Difference (%D):

$$\%D = \frac{\text{Average RRF from initial curve RRF cpd.}}{\text{Average RRF from initial curve}} \times 100$$

11.6 Calculation for Determining Concentration of Compounds:

$$\text{Conc. Cpd(ppbv)} = \frac{\text{Area Cpd.in sample}}{\text{Area I.S. in sample}} \times \frac{\text{Conc. I.S.}}{\text{RRF daily std.}} \times \text{Dil. Factor (See 11.2)}$$

The area of the primary quantitation ion is used in the calculation
I.S.= Internal Standard

11.7 Calculation for Percent Recovery (%Rec):

$$\%Rec \quad \frac{\text{Amount cpd. recovered}}{\text{Amount cpd. spiked}} \times 100$$

11.8 Calculation for Relative Percent Difference (RPD):

$$RPD = \frac{\text{Value A - Value B}}{\text{Average of Values}} \times 100$$

12. Reporting

12.1 Reporting units are ppbv. If results are to be reported in ng/L or ug/m^3 use the following equation:

$$\text{result ppbv} \times \frac{\text{Molecular weight of compound}}{24.5} = ng/L = ug/m^3$$

Note: 24.5 is the volume of ideal gas at 25 degrees Centigrade and 1 atm.

12.2 Reporting limits

See Table 12. All reporting limits and MDLs must be derived on GC/MS systems at the Enseco Air Toxics laboratory and are periodically updated. Analytes that are detected below the Reporting Limit and above the Method Detection Limit are not routinely reported. When project requirements or Data Quality Objectives specify reporting such values they will be identified and given an estimated concentration based on the following procedure:

12.2.1 Retention time criteria must be met.

12.2.2 Mass spectral criteria must be met. At a minimum, the quantitation ion and two confirmatory ions must be present. (A quantitation ion and one confirmatory ion must be present for chloromethane, chloroethane, vinyl chloride and acetone). All ions must be resolved from background and noise.

12.2.3 Values must be greater than the MDL and less than the RL.

12.2.4 All estimated values must be reported with a "J" footnote qualifier.

12.2.5 The paragraph in section 9.1.1.3 may be applied when necessary.

12.3 Significant figures

All results should be reported to two significant figures.

12.4 No conversion of the analytical results to the standard conditions is made.

13. References

13.1 Method Source

"EPA Compendium Method TO-14. The Determination of Volatile Organic Compounds (VOCs) in Ambient Air using SUMMA Passivated Canister Sampling and Gas Chromatographic Analysis."

13.2 Deviations from Method

13.2.1 Helium is used for dilution purposes.

13.2.2 TO-14 recommends the use of a .32 mm column coupled directly to the MSD. With the HP system, the MSD can only handle flow of 1 mL/min or less. The .32 mm column provides ~ 3 mL/min. Enseco uses a .53 mm column through a jet separator.

13.2.3 TO-14 describes an inlet system that uses a vacuum to pull a slip stream sample through the trap. Enseco uses the pressure of the sample canister to drive the sample through the trap.

13.2.4 TO-14 describes the use of a Nafion dryer to remove excess moisture from air matrices. Enseco does not use a Nafion dryer since polar compounds may be lost during this removal step.

13.2.5 TO-14 describes the use of SUMMA passivated steel canisters for sampling and analysis. No mention is made of Tedlar sampling bags. Enseco analyzes samples in Tedlar bags for VOCs using the same procedures described herein. A modification to the method is noted on the final report.

TABLE 11. Concentration of Daily Check Standard

	Compound	Concentration (ppbv)
2)	Dichlorodifluoromethane (Freon 12)	45.42
3)	Chloromethane	43.84
4)	1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon 114)	42.50
5)	Vinyl chloride	44.74
6)	Bromomethane	44.74
7)	Chloroethane	42.96
8)	Trichlorofluoromethane (11)	42.06
9)	1,1-Dichloroethene	48.32
10)	Carbon disulfide	54.72
11)	1,1,2-Trichloro- 1,2,2-trifluoroethane (Freon 113)	44.30
12)	Acetone	44.30
13)	Methylene chloride	48.32
14)	trans-1,2-Dichloroethene	48.32
15)	1,1-Dichloroethane	47.42
16)	Vinyl Acetate	37.50
17)	cis-1,2-Dichloroethene	47.88
18)	2-Butanone	45.64
19)	Chloroform	47.82
20)	1,1,1-Trichloroethane	44.30
21)	Carbon tetrachloride	45.20
23)	Benzene	47.82
24)	1,2-Dichloroethane	49.22
25)	Trichloroethene	36.68
26)	1,2-Dichloropropane	48.32
27)	Bromodichloromethane	48.32
28)	cis-1,3-Dichloropropene	46.08
29)	4-Methyl-2-pentanone	48.76
30)	Toluene	48.32
32)	trans-1,3-Dichloropropene	53.70
33)	1,1,2-Trichloroethane	53.70
34)	Tetrachloroethene	51.00
35)	2-Hexanone	52.80
36)	Dibromochloromethane	50.56
37)	1,2-Dibromoethane	44.30
38)	Chlorobenzene	48.32
39)	Ethylbenzene	53.70
40)	1,4-and 1,3-(p,m) Xylene	103.36

TABLE 11 cont. Concentration of Daily Check Standards

	Compound	Concentration (ppbv)
41)	1,2-(ortho) Xylene	50.12
42)	Styrene	55.92
43)	Bromoform	37.58
44)	1,1,2,2-Tetrachloroethane	55.48
45)	Benzyl chloride	32.98
46)	4-Ethyltoluene	39.82
47)	1,3,5-Trimethylbenzene	42.50
48)	1,2,4-Trimethylbenzene	41.16
49)	1,3-Dichlorobenzene	34.36
50)	1,4-Dichlorobenzene	46.54
51)	1,2-Dichlorobenzene	55.92
52)	1,2,4-Trichlorobenzene	40.18
53)	Hexachlorobutadiene	35.98

TABLE 12. VOC Reporting Limits

	Compound	Reporting Limits (ppbv)
2)	Dichlorodifluoromethane (Freon 12)	2.0
3)	Chloromethane	4.0
4)	1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon 114)	2.0
5)	Vinyl chloride	2.0
6)	Bromomethane	2.0
7)	Chloroethane	4.0
8)	Trichlorofluoromethane (11)	2.0
9)	1,1-Dichloroethene	2.0
10)	Carbon disulfide	10
11)	1,1,2-Trichloro- 1,2,2-trifluoroethane (Freon 113)	2.0
12)	Acetone	10
13)	Methylene chloride	2.0
14)	trans-1,2-Dichloroethene	2.0
15)	1,1-Dichloroethane	2.0
16)	Vinyl Acetate	10
17)	cis-1,2-Dichloroethene	2.0
18)	2-Butanone	10
19)	Chloroform	2.0
20)	1,1,1-Trichloroethane	2.0
21)	Carbon tetrachloride	2.0
23)	Benzene	2.0
24)	1,2-Dichloroethane	2.0
25)	Trichloroethene	2.0
26)	1,2-Dichloropropane	2.0
27)	Bromodichloromethane	2.0
28)	cis-1,3-Dichloropropene	2.0
29)	4-Methyl-2-pentanone	4.0
30)	Toluene	2.0
32)	trans-1,3-Dichloropropene	2.0
33)	1,1,2-Trichloroethane	2.0
34)	Tetrachloroethene	2.0
35)	2-Hexanone	4.0
36)	Dibromochloromethane	2.0
37)	1,2-Dibromoethane	2.0
38)	Chlorobenzene	2.0
39)	Ethylbenzene	2.0
40)	1,4-and 1,3-(p,m) Xylene	2.0

TABLE 12 cont. VOC Reporting Limits

	Compound	Reporting Limits (ppbv)
41)	1,2-(ortho) Xylene	2.0
42)	Styrene	2.0
43)	Bromoform	2.0
44)	1,1,2,2-Tetrachloroethane	2.0
45)	Benzyl chloride	2.0
46)	4-Ethyltoluene	2.0
47)	1,3,5-Trimethylbenzene	2.0
48)	1,2,4-Trimethylbenzene	2.0
49)	1,3-Dichlorobenzene	2.0
50)	1,4-Dichlorobenzene	2.0
51)	1,2-Dichlorobenzene	2.0
52)	1,2,4-Trichlorobenzene	4.0
53)	Hexachlorobutadiene	4.0

TABLE 13. VOC Key Ions

	Compound	Primary	Secondary
1)	Bromochloromethane I.S.#1	49	130,128
2)	Dichlorodifluoromethane (Freon 12)	85	87, 50
3)	Chloromethane	50	52
4)	1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon 114)	85	135, 87
5)	Vinyl chloride	62	64
6)	Bromomethane	94	96, 79
7)	Chloroethane	64	66, 49
8)	Trichlorofluoromethane (11)	101	103, 66
9)	1,1-Dichloroethene	61	96, 63, 98
10)	Carbon disulfide	76	78, 44
11)	1,1,2-Trichloro- 1,2,2-trifluoroethane (Freon 113)	101	151,103, 85
12)	Acetone	43	58
13)	Methylene chloride	49	84, 86
14)	trans-1,2-Dichloroethene	61	96, 98, 63
15)	1,1-Dichloroethane	63	65, 83
16)	Vinyl Acetate	43	44, 86, 42
17)	cis-1,2-Dichloroethene	61	96, 98, 63
18)	2-Butanone	72	43, 57
19)	Chloroform	83	85, 47
20)	1,1,1-Trichloroethane	97	99, 61
21)	Carbon tetrachloride	117	119,121, 82
22)	1,4-Difluorobenzene I.S.#2	114	63, 88
23)	Benzene	78	50, 52, 77
24)	1,2-Dichloroethane	62	64, 49, 98
25)	Trichloroethene	130	95,132, 97
26)	1,2-Dichloropropane	63	62, 41, 39
27)	Bromodichloromethane	83	85,129
28)	cis-1,3-Dichloropropene	75	77, 39
29)	4-Methyl-2-pentanone	43	58,100, 85
30)	Toluene	91	65, 92
31)	Chlorobenzene-d5 I.S. #3	117	52, 54, 82
32)	trans-1,3-Dichloropropene	75	77, 39
33)	1,1,2-Trichloroethane	97	83, 85, 61
34)	Tetrachloroethene	166	129,131,164
35)	2-Hexanone	43	58, 57,100
36)	Dibromochloromethane	129	127,208,131
37)	1,2-Dibromoethane	107	109,188
38)	Chlorobenzene	112	77,114
39)	Ethylbenzene	91	106, 65, 51
40)	1,4-and 1,3-(p,m) Xylene	91	106,105, 77

TABLE 13 cont. VOC Key Ions

	Compound	Primary	Secondary
0			
41)	1,2-(ortho) Xylene	91	106,105, 77
42)	Styrene	104	78,103, 51
43)	Bromoform	173	171,175, 93
44)	1,1,2,2-Tetrachloroethane	83	85,133,131
45)	Benzyl chloride	91	126, 63
46)	4-Ethyltoluene	105	120, 77
47)	1,3,5-Trimethylbenzene	105	120, 77
48)	1,2,4-Trimethylbenzene	105	120, 77
49)	1,3-Dichlorobenzene	146	148,111, 75
50)	1,4-Dichlorobenzene	146	148,111, 75
51)	1,2-Dichlorobenzene	146	148,111, 75
52)	1,2,4-Trichlorobenzene	180	182,109,145
53)	Hexachlorobutadiene	225	227,223